

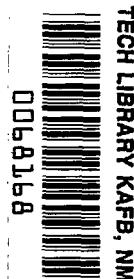
**NASA
Technical
Paper
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Tricresylphosphate (TCP)**

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**Mechanism of Lubrication by
Tricresylphosphate (TCP)**

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*Lewis Research Center
Cleveland, Ohio*



National Aeronautics
and Space Administration

Scientific and Technical
Information Branch

Summary

The coefficient of friction was measured as a function of temperature on a pin-on-disk tribometer. Pins and disks of 440C and 52100 steels were lubricated with tricresylphosphate (TCP), 3.45 percent TCP in squalene, and pure squalene. M-50 pins and disks were lubricated with 3.45 percent TCP in squalene and pure squalene. Experiments were conducted under limited lubrication conditions in dry (<100 ppm H_2O) air and dry (<20 ppm H_2O) nitrogen at 50 rpm (equivalent to a sliding velocity of 13 cm sec^{-1}) and a constant load of 9.8 N (1 kg). Characteristic temperatures T_f were identified for TCP on 52100 steel and for squalene on M-50 and 52100 steels, where the friction decreased because of a chemical reaction between the lubricant and the metal surface. The behavior of squalene obscured the influence of 3.45 percent TCP solute on the friction of the system. Wear volume measurements demonstrated that wear was lowest at temperatures just above T_f . Comparison of the behavior of TCP on M-50, 440C, and 52100 steels revealed that the TCP either reacted to give T_f behavior or produced initial failure in the temperature range $223^\circ \pm 5^\circ \text{C}$. The 440C steel yielded a peak in friction; the 52100 and M-50 steels had assignable T_f values in this temperature range. Oxygen was essential for the reaction of TCP with the metal surface.

Introduction

The mechanism by which tricresylphosphate (TCP) functions as an antiwear, extreme-pressure lubricant has been studied since 1940, when Beeck, Givens, and Williams (ref. 1) proposed the formation of a eutectic layer of iron and iron phosphide. Twenty-five years later several workers demonstrated that this idea was not tenable. Barcroft and Daniels (ref. 2) proposed the existence of a phosphate layer; Godfrey (ref. 3) demonstrated the presence of FePO_4 and $\text{FePO}_4 \cdot 2\text{H}_2\text{O}$ on the surface of TCP-lubricated steel by electron diffraction; Bieber, Tewksbury, and Klaus (ref. 4) addressed the source of the phosphate by proposing that reactive acidic and polar impurities in commercial TCP are responsible for the formation of the phosphate layer. Current opinion is that phosphate is indeed formed on the surface of metals under a variety of conditions (refs. 5 to 9).

The coefficients of friction for lubricants containing TCP have been measured under a variety of conditions (refs. 9 to 12) but only once (ref. 9) over a sufficient temperature range to achieve failure of the lubricant. Faut and Wheeler demonstrated the presence of a characteristic temperature for TCP lubricating M-50 steel, where the lubricant reacted with the steel to produce a phosphate layer on the metal (ref. 9). The

present investigation was conducted to determine the chemical reactions of the lubricant with other steels. Therefore the coefficients of friction were measured for TCP lubricating 52100 and 440C steels. In addition, a 3.45 percent TCP solution in squalene was used to measure friction with the three steels: 440C, 52100, and M-50. Wear studies were conducted to further delineate the effect of the phosphate layer.

Experimental Procedure

Coefficients of friction were measured as a function of temperature by using a pin-on-disk tribometer equipped with an induction heater for the disk (fig. 1). The details of the experimental procedure are given in reference 9. All experiments in the present report were conducted under limited lubrication conditions (i.e., approx 1 cm^3 of lubricant was placed directly on the rotating disk). Lubricants used for nitrogen atmosphere experiments were degassed and dehydrated as described in reference 9. The base fluid for preparation of the TCP solution was 2,6,10,15,19,23-hexamethyltetracosane, commonly known as squalene. The 3.45 percent TCP in squalene was prepared from commercial laboratory reagent-grade squalene and TCP. The commercial TCP had been prepared from an 80 percent para-20 percent meta mixture of cresols.

Wear studies were conducted by setting the temperature of the disk at a fixed level and then stabilizing the temperature with air or nitrogen flow. When the temperature of the disk was stabilized, the lubricant was placed on the rotating disk and the pin was loaded to 9.8 N (1 kg) for a fixed time. The sliding velocity was set at 50 rpm (equivalent to a sliding velocity of 13 cm sec^{-1}) for each experiment. After the experiment was concluded, the pins were removed, washed carefully with Freon to remove traces of the lubricant, and examined microscopically. Photographs were taken of the wear scars on the pins, and the wear scar diameters were determined from the photographs.

The air used contained less than 100-ppm water. The nitrogen used contained less than 20-ppm water. Smoothed friction-temperature curves are presented in this report. Primary data curves and the use of smoothed curves are discussed in reference 9. This same reference includes a discussion of the uncertainties in the temperature measurements.

The hardness of each pin and disk was measured before and after each experiment. It is well known that friction can change as the hardnesses of the metals change (ref. 13). Therefore only those results are presented in which the hardnesses changed by less than 10 percent during the course of the experiment. Indeed, the experiments that required heating the disks to

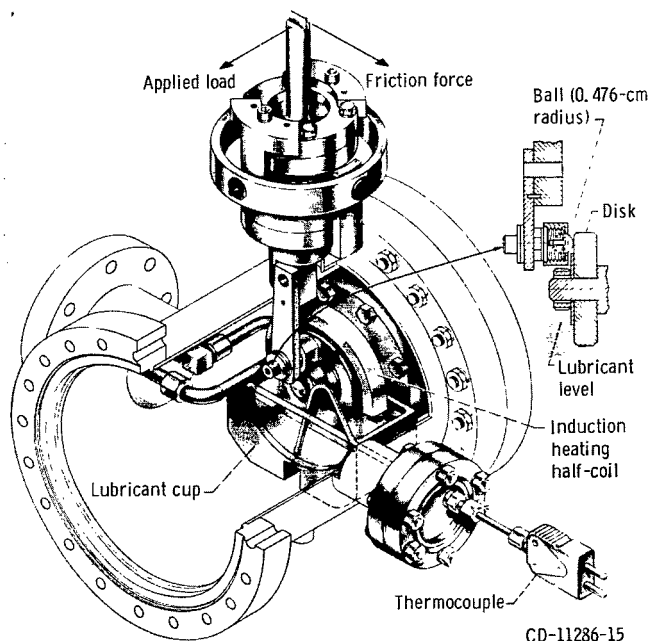


Figure 1. – Pin-on-disk tribometer equipped with an induction heater for temperature-variation studies of friction.

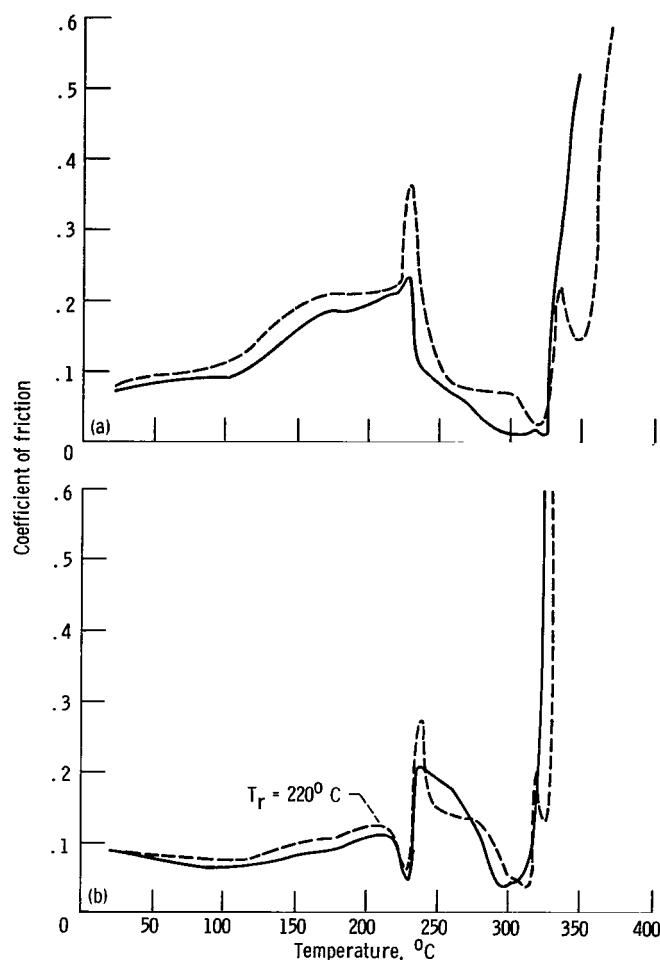
approximately 700° C in order to remove or react adsorbed oxygen could not be performed for 52100 steel because of the great variation in hardness with temperature (ref. 14).

Experimental Results

The results of the friction-temperature measurements are presented in figures 2 to 7. Table I summarizes the experiments presented in this report.

Pure TCP

The friction-temperature curves for 440C steel lubricated by pure TCP in dry air are shown in figure 2(a) and exhibit a peak in friction at 225° to 230° C for the two experiments. After the peak the friction decreased before failure, which occurred at temperatures as low as 325° C. The friction-temperature behavior of 440C steel can be compared with that of 52100 steel in figure 2(b). For 52100 steel the friction underwent a pronounced decrease beginning at 220° C and reached a minimum at 230° C. This minimum was followed by a peak in friction at 320° to 330° C. The minimum beginning at 220° C is attributed to the characteristic temperature T_r and is associated with a chemical reaction between the lubricant and the metal surface (refs. 9 and 15). Note particularly that no T_r value can be assigned to the 440C steel curve since no sharp decrease in friction occurred before the peak at 225° C.



(a) 440C Pins sliding against 440C disks.

(b) 52100 Pins sliding against 52100 disks.

Figure 2. – Coefficient of friction as a function of temperature for 440C and 52100 steels under limited lubrication by pure TCP in dry air. Each curve represents an independent experiment.

TABLE I. – SUMMARY OF EXPERIMENTS PERFORMED

Steel used	Lubricant used	Environment
440C	Pure TCP	Dry air
52100	Pure TCP	Dry air
440C	Pure TCP	Dry nitrogen
52100	Pure TCP	Dry nitrogen
440C	Pure squalene	Dry air
52100	Pure squalene	Dry air
M-50	Pure squalene	Dry air
440C	Pure squalene	Dry nitrogen
M-50	Pure squalene	Dry nitrogen
440C	3.45 Percent TCP in squalene	Dry air
52100	3.45 Percent TCP in squalene	Dry air
M-50	3.45 Percent TCP in squalene	Dry air
M-50	3.45 Percent TCP in squalene	Dry nitrogen

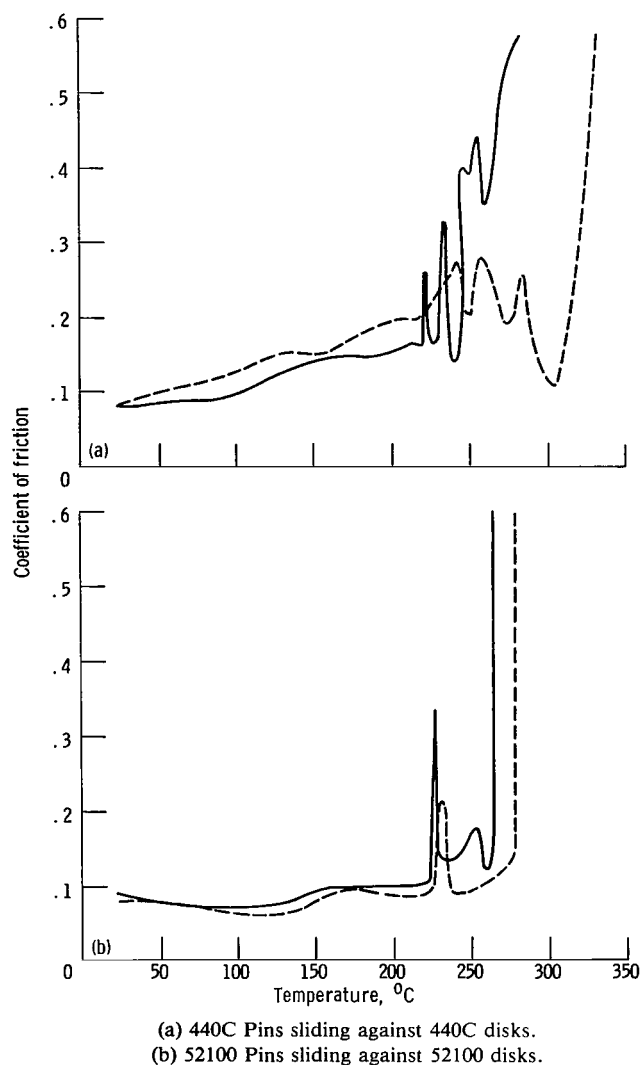


Figure 3. – Coefficient of friction as a function of temperature for 440C and 52100 steels under limited lubrication by pure TCP in dry nitrogen. Each curve represents an independent experiment.

Figures 3(a) and (b) present the coefficient of friction as a function of temperature for 440C and 52100 steels, respectively, lubricated with pure TCP in a dry nitrogen atmosphere. The 440C curves exhibit multiple peaks in friction beginning at 220° C. The 52100 curves show initial sharp peaks in friction at approximately 230° C. It is interesting that these peaks occur in the same temperature range as the T_r values obtained for 52100 steel with TCP in dry air. It is also noteworthy that 440C steel begins its multiple peak behavior near the temperature where the peak is found for 440C in dry air.

Squalene

The great majority of published work involving TCP deals with solutions in which TCP is a minor component (i.e., the solute). The coefficient of friction has not been

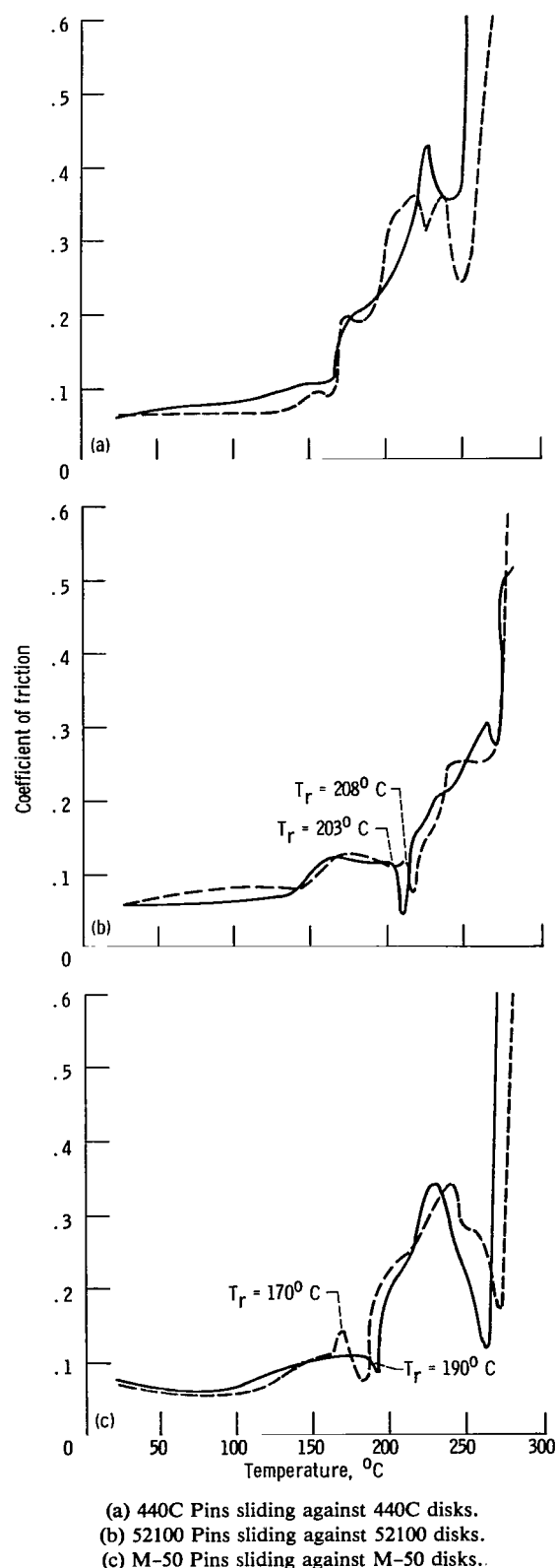


Figure 4. – Coefficient of friction as a function of temperature for 440C, 52100, and M-50 steels under limited lubrication by squalene in dry air. Each curve represents an independent experiment.

studied as a function of temperature over large temperature ranges for such solutions. To determine the behavior of TCP solutions, we chose a single compound as the solvent, one whose frictional behavior as a function of temperature could be carefully characterized. Figures 4(a) to (c) present friction-temperature curves for 440C, 52100, and M-50 steels, respectively, lubricated with pure squalene in dry air. The 440C curves in figure 4(a) contain no easily assignable T_f values, but the 52100 and M-50 steel curves do exhibit assignable values. The 52100 steel shows sharp decreases in friction at 203° and 208° C in figure 4(b). These temperatures were assigned as the T_f values for squalene lubricating 52100 steel in dry air. The M-50 behavior in figure 4(c) is similar, with T_f values assigned as 170° and 190° C.

Figures 5(a) and (b) present friction-temperature curves for squalene on 440C and M-50 steels, respectively, in dry nitrogen. Characteristic temperatures were assigned for these steels as follows: 440C as 155° and 160° C, and M-50 as 170° C for both curves.

3.45 Percent TCP in Squalene

Figure 6 presents friction-temperature curves for 3.45 percent TCP in squalene in dry air. Again the 440C steel (fig. 6(a)) exhibits multiple peaks without a significant decrease in friction assignable to T_f . These multiple peaks occur in the temperature range 150° to 180° C, the same range where friction increased for pure squalene on 440C in dry air. In effect, the 3.45 percent TCP seems to have no effect on the friction of 440C steel in dry air. In contrast, 52100 steel (fig. 6(b)) exhibits a decrease in friction at assignable T_f values of 205° and 212° C. This is in the same temperature range as squalene alone but is also only about 10 degrees below the T_f values for TCP alone on 52100 steel in dry air (fig. 2(b)). It seems reasonable to suggest that the 3.45 percent TCP in squalene had no effect on the friction experienced by the 52100 steel in dry air, but the proximity of the pure-TCP T_f values makes this suggestion a tentative one. The M-50 steel (fig. 6(c)) exhibits a decrease in friction similar to that observed for squalene alone in figure 4(c) (i.e., T_f values of 175° to 180° C). These T_f values are in the same temperature range as those found for pure squalene and well below the T_f values found for pure TCP (fig. 5(b)). This suggests that the 3.45 percent TCP has no observable effect on the friction experienced by M-50 steel. Figure 7 presents the curves for 3.45 percent TCP in squalene on M-50 steel in dry nitrogen. No T_f values were assignable and failure temperatures were 162° and 169° C. These temperatures matched very well with those for squalene alone (fig. 5(b)), an indication that the TCP did not affect the friction in this experiment.

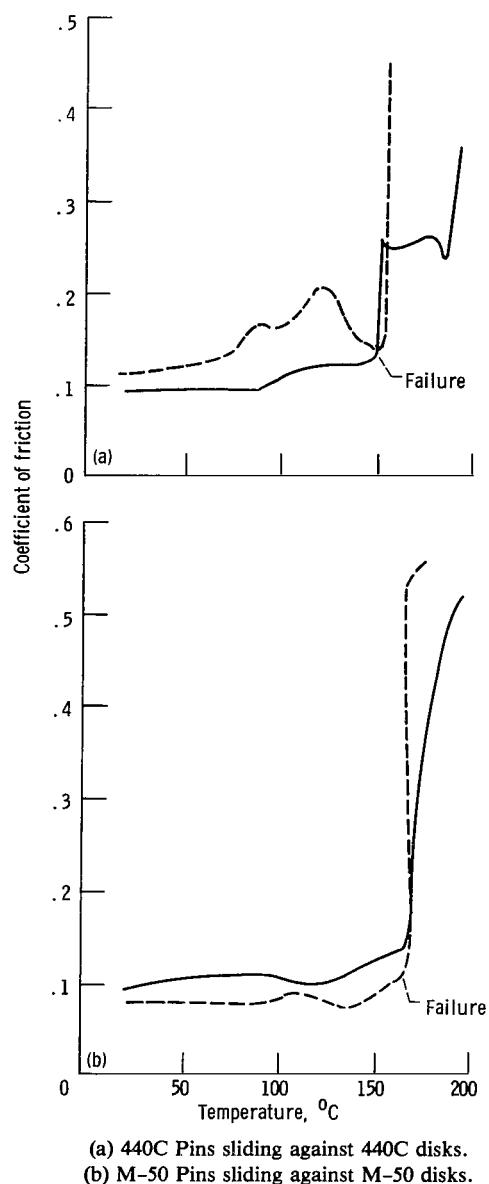
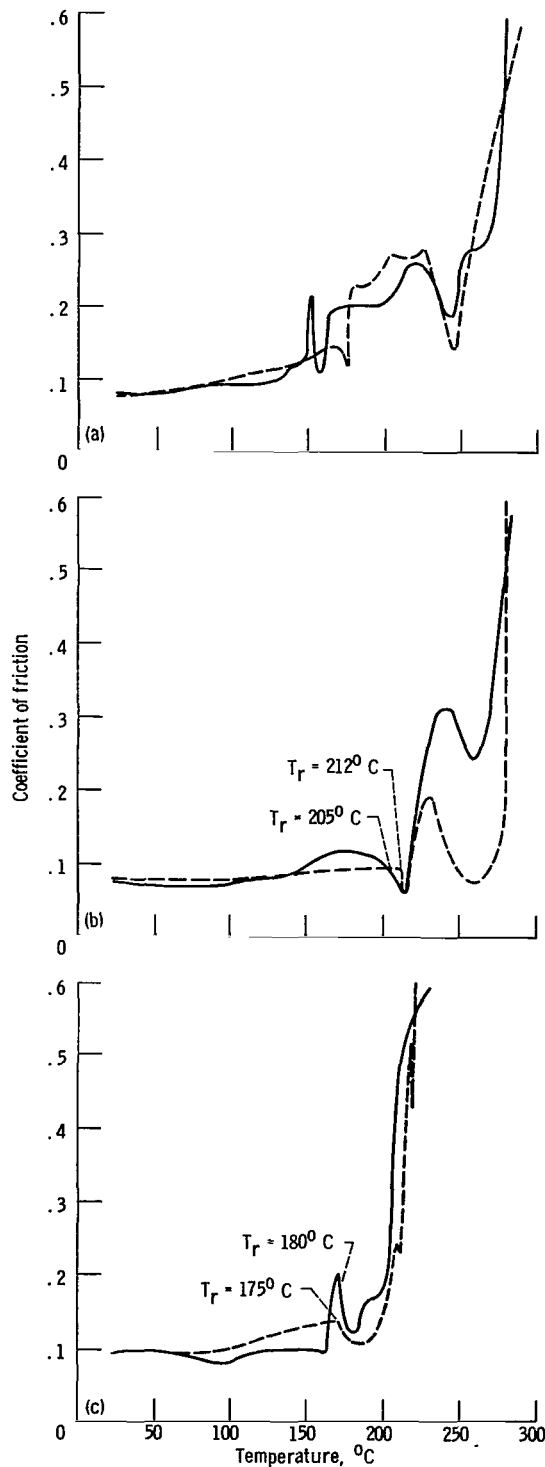


Figure 5. — Coefficient of friction as a function of temperature for 440C and M-50 steels under limited lubrication by squalene in dry nitrogen. Each curve represents an independent experiment.

Wear Volumes

Wear volumes for 52100 and 440C steels lubricated with TCP were determined. They are summarized in table II. The photographs from which the data in table II were gathered are presented in figures 8 and 9.

The temperatures used for the wear analysis were chosen on the basis of the friction-temperature curves in figures 2(a) and (b). The low temperature ranges were selected as representative of the lubrication before any dramatic change in the friction. The middle temperature ranges were chosen to be as close as possible to the first



(a) 440C Pins sliding against 440C disks.
 (b) 52100 Pins sliding against 52100 disks.
 (c) M-50 Pins sliding against M-50 disks.

Figure 6. — Coefficient of friction as a function of temperature for 440C, 52100, and M-50 steels under limited lubrication by 3.45 percent TCP in squalene in dry air. Each curve represents an independent experiment.

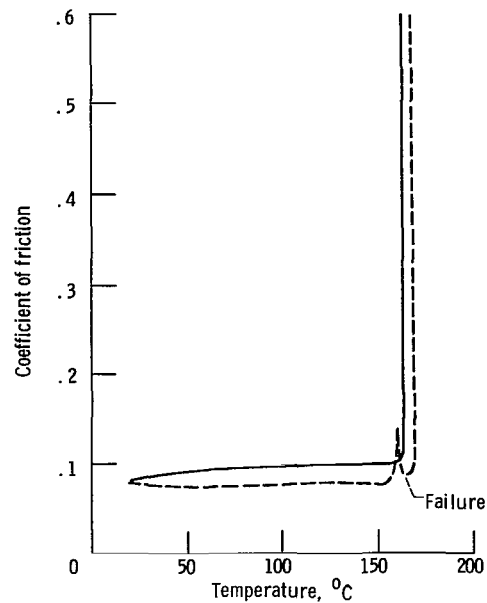


Figure 7. — Coefficient of friction as a function of temperature for M-50 pins sliding against M-50 disks under limited lubrication by 3.45 percent TCP in squalene in dry nitrogen. Each curve represents an independent experiment.

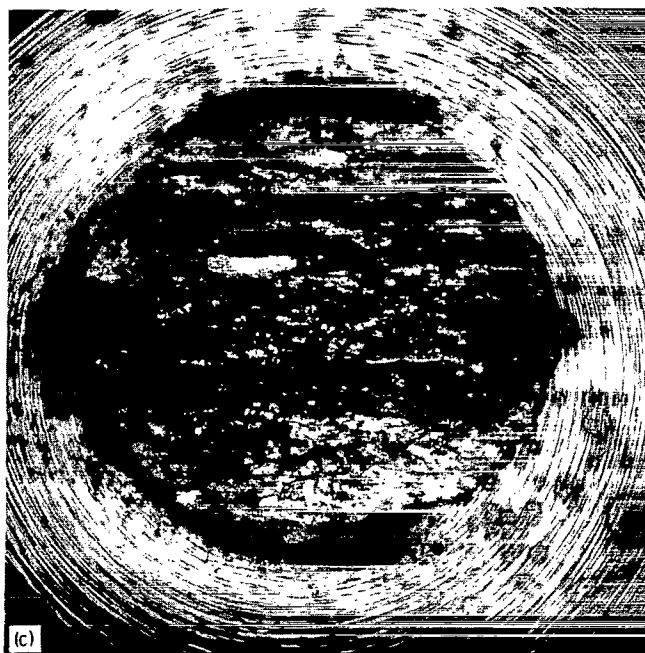
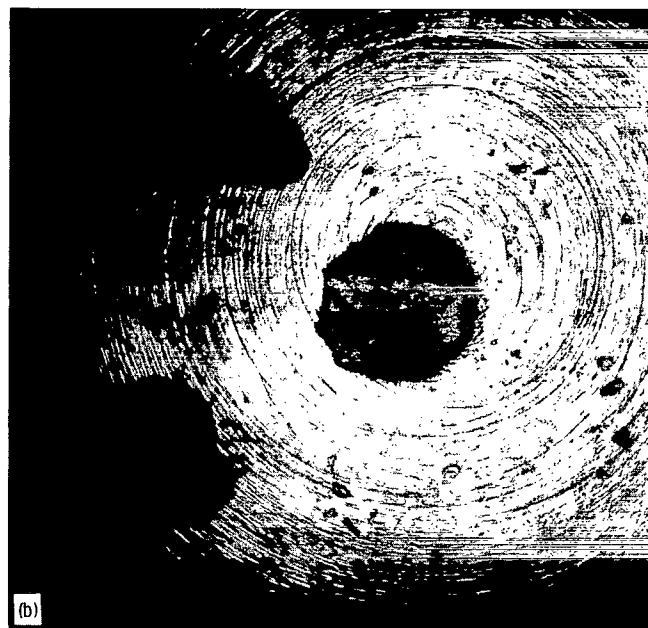
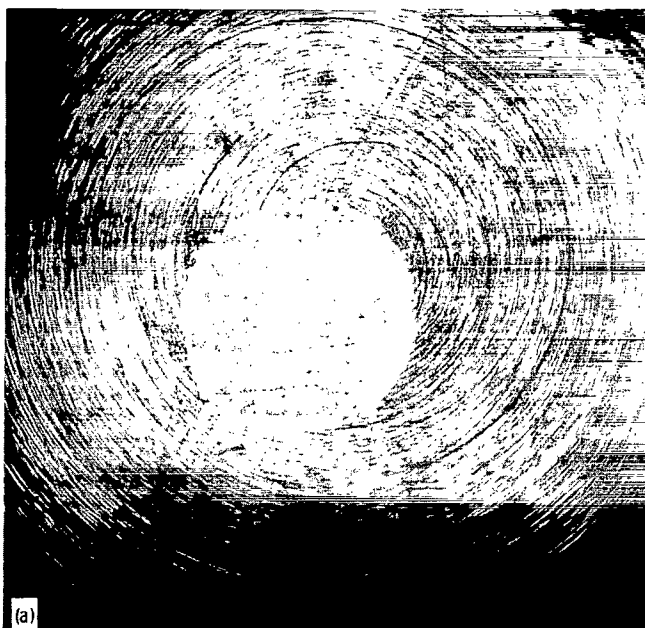
TABLE II. — WEAR VOLUMES
 USING PURE TCP IN DRY AIR^a

Steel used	Temperature range, °C	Wear volume, cm ³
52100	168-172	55×10^{-8}
	229-231	14
	329-336	1300
440C	167-170	120
	234-237	6.3
	353-357	4700

^aEach experiment was run for 15 min.

minimum in friction (52100 steel) or just past the first peak in friction (440C steel). The high temperature ranges represent the failure portion of the curves. For both 52100 and 400C steel samples the lowest wear occurred in the middle temperature range and the highest wear at the failure temperatures.

The photographs of the wear scars show more severe striations in the 52100 steel at the low temperatures than occurred in the 440C steel. The 52100 sample also had some evidence of material transfer. The highest temperature photographs show the most severe wear for both 52100 and 440C steels.



(a) 168° to 172° C.
(b) 229° to 231° C.
(c) 329° to 336° C.

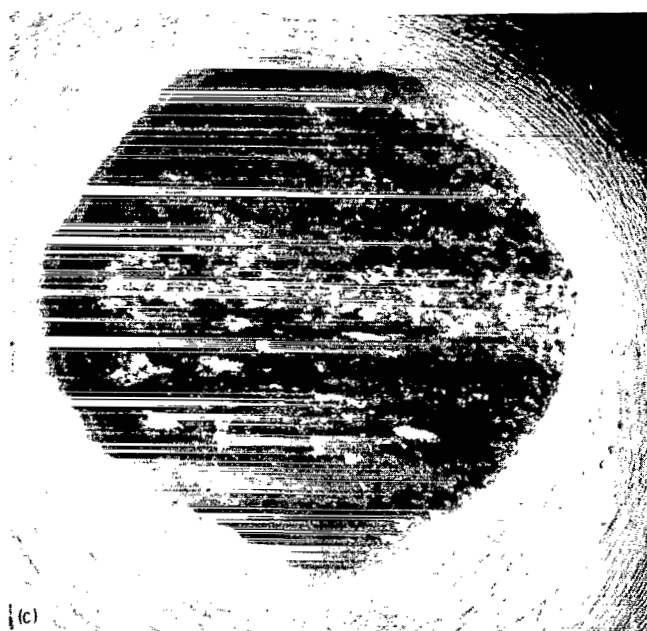
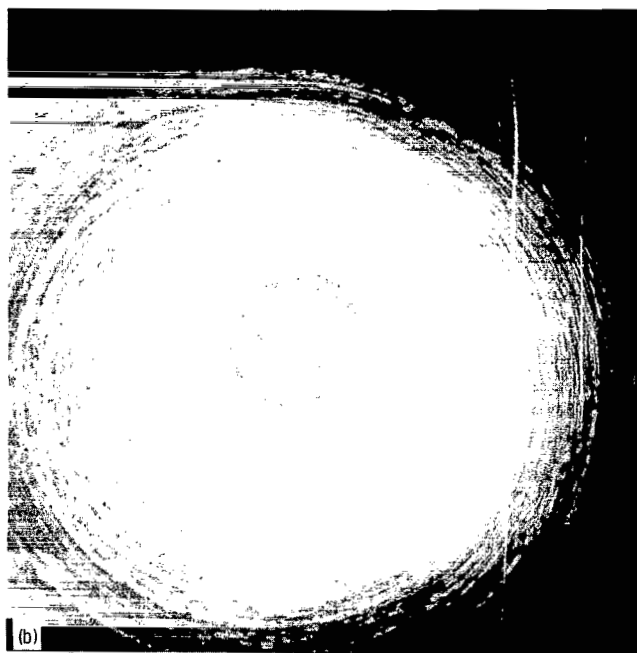
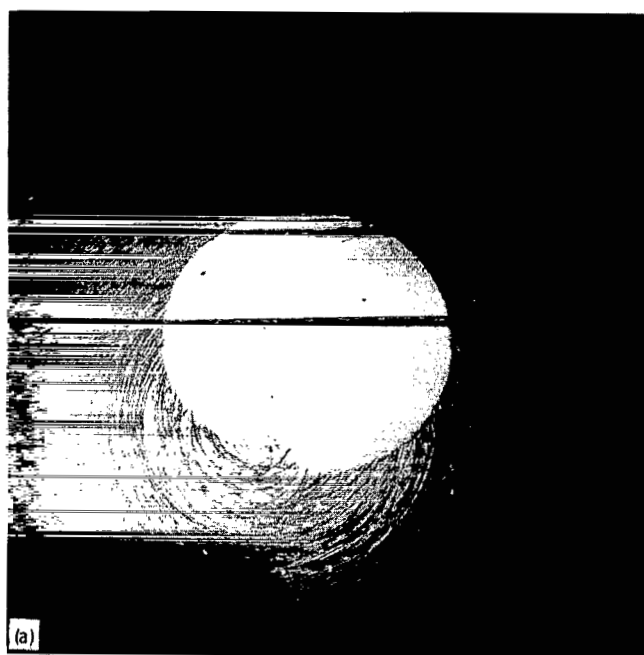
Figure 8. — Photomicrographs of wear areas on 52100 pins after sliding against TCP-lubricated 52100 disks for 15 min in temperature ranges indicated.

Discussion of Experimental Results

One of the most striking features of the friction-temperature studies using pure TCP is the persistent occurrence of either failure temperatures or characteristic temperatures associated with a chemical reaction on the

surface T_r in the temperature range 220° to 230° C. Table III presents these data for the three steels.

In each case noted in table III the frictional behavior was smooth and regular until the temperature reached the 220° C range. At this point the friction either decreased at a characteristic temperature T_r or failure occurred.



- (a) 167° to 170° C.
 (b) 234° to 237° C.
 (c) 353° to 357° C.

Figure 9. — Photomicrographs of wear areas on 440C pins after sliding against TCP-lubricated 440C disks for 15 min in temperature ranges indicated.

Faut and Wheeler (ref. 9) presented evidence that a chemical reaction occurs between TCP and M-50 steel to form a phosphate at T_r . It will be useful to compare the behavior of M-50 steel lubricated by TCP in dry air with the behavior of 440C and 52100 steels under the same

conditions. The M-50 steel curves show a pronounced decrease in friction beginning at T_r . Any increase in friction occurring before final failure reached a maximum at friction values no higher than those observed before T_r . The 52100 steel curves exhibit a less

TABLE III. - COMPARISON OF CHARACTERISTIC OR FAILURE TEMPERATURES FOR THREE STEELS LUBRICATED BY PURE TCP

Steel	Atmosphere	Characteristic temperature, T_r , °C	Failure temperature, °C	Comments
M-50	Dry air	225	---	Reference 9
	Dry N ₂	215	---	Reference 9
	Dry N ₂	---	218	Reference 9 (preheated)
440C	Dry air	---	---	Peaks at 225° to 230° C
	Dry N ₂	---	220	Multiple peaks
52100	Dry air	220	---	-----
	Dry N ₂	---	---	Peaks at 227° and 231° C

pronounced decrease in friction beginning at T_r and a peak in friction immediately following the minimum due to T_r . The 440C steel curves exhibit only the large peak and no minimum before the peak. This suggests that 52100 steel reacted with TCP and that the adsorbed lubricant film began to fail before the chemical reaction between the TCP and the metal surface was complete. For 440C steel the adsorbed lubricant film appeared to fail before the chemical reaction occurred to form the protective film. In effect, the chemical reactivity of TCP toward these steels seems to be of the order M-50 > 52100 > 440C. The latter two are in the order suggested by previous investigators (refs. 6 and 7).

Comparing the friction behavior for TCP lubricating 440C and 52100 steels in dry air and in dry nitrogen confirmed the essential role of oxygen in forming the protective film at T_r . The dry air experiments produced assignable T_r values because a protective film formed on the metal surface, but the dry nitrogen experiments produced failure behavior in the same temperature range. The 440C steel also exhibits the effects of oxygen in figures 2 and 4. The dry air experiment with 440C steel produced a decrease in friction after the initial peak at 227° C, but the dry nitrogen experiments produced no such decrease.

The TCP did appear to form a protective film with each steel. For M-50 and 52100 steels this layer began to form at T_r . For 440C steel there was no T_r , only a failure in the same range. However, because the wear volume analysis indicated minimum wear just past the failure temperature, 440C steel also may form a protective film. The protective films were formed in the dry air atmosphere, not in the nitrogen atmosphere. Indeed the wear for both 52100 and 440C steels was significantly less at temperatures slightly above 230° C than at the lowest temperature or the failure temperature.

On the basis of the behavior of TCP on M-50 steel, it has been suggested (ref. 9) that 220° C is the characteristic temperature of the antiwear action of TCP. When the results of the experiments with 52100 and 440C steels were added to this analysis, a mean temperature of

223° C with a standard deviation of 5 degrees was found. Therefore under limited lubrication conditions TCP will react with steel surfaces at 223° ± 5° C. The use of this temperature for the limiting usefulness of TCP will vary with the hardness of the metal. All of the data used for temperature calculations were from steels with hardness R_c between 55 and 60.

Friction can also vary with composition. It is reasonable to expect that the chemical reaction on the metal surface will be with iron, the major component of the steels. The 440C steel is the most questionable in this regard because it has a high chromium content. However, Ferrante (ref. 16) has shown that the surface of 440C steel is composed of iron oxides up to about 700° C. (After 700° C the chromium oxides become the major surface material.) Therefore we can expect any chemical reaction between TCP and the steels to be essentially a reaction with iron or iron oxides.

The friction-temperature behavior for squalene lubricating 52100 and M-50 steels in air exhibits assignable T_r values. If these temperatures are correctly assigned to the occurrence of a chemical reaction between the lubricant and the metal surface, some type of reaction must also occur between the squalene and the steel surface. It is well known (refs. 17 and 18) that hydrocarbon lubricants can react with oxygen to form a variety of compounds including carbonyls and alcohols. The squalene has six tertiary hydrogen atoms in its structure that will be particularly reactive as compared with secondary and primary hydrogen atoms (ref. 19). Therefore reaction with oxygen should be easier for squalene than for unsubstituted straight-chain hydrocarbon lubricants. The presence of assignable T_r values is most likely due to oxidation products of squalene reacting with the steel surfaces. The 440C steel was not sufficiently reactive to allow measurable reaction; this is in accord with the behavior already discussed for TCP.

The lubrication by squalene in dry nitrogen atmosphere did not result in measurable T_r values, as would be expected if oxidation products were reacting with the steel.

When TCP is used as an additive in liquid lubricant systems, it is usually present in concentrations below 10 percent, frequently less than 5 percent. Using TCP as an additive reduces wear (refs. 5, 10, 11, 20, and 21), but not friction (refs. 10 and 12) although Wiegand and Broszeit (ref. 11) have observed a reduction in friction as a function of load for 1 percent TCP in paraffin. The presence of 3.45 percent TCP in squalene did not result in any significant difference in friction from that with squalene alone. The ability of the oxidation products to react with the metal surface apparently obscured any reaction by the smaller amount of solute (TCP). The observations of Weigand and Broszeit are not in conflict with this suggestion if the paraffin solvent used by them contained no tertiary hydrogen atoms. The secondary hydrogen atoms necessarily present in any unsubstituted straight-chain hydrocarbon molecules react with greater difficulty than the tertiary hydrogen atoms. The reactivity of TCP would then be stronger than the reactivity of oxidation products of the solvent and thereby produce a decrease in the friction.

The TCP mechanism suggested by the preceding analysis can be summarized as follows: TCP is adsorbed onto the surface of the steel. The adsorbed layer undergoes limited reaction with the surface until T_r is attained. Then the TCP is either desorbed, leading to a failure behavior as in 440C steel, or reacts with the surface, leading to a decrease in friction and an assignable T_r value, as with M-50 and 52100 steels. The reaction with 52100 steel is sufficiently limited to allow the desorption of the TCP from the reacted layer to influence the friction (i.e., to produce the peak in friction immediately after the T_r behavior). The TCP continues to react with the surface to form a protective coating. Failure occurs when the liquid film is exhausted and the protective layer has worn through.

The fact that T_r occurs within a very narrow temperature range for the three steels points to a lack of sensitivity by the TCP for the surfaces. However, it is known that all three steels present iron oxide surfaces to the lubricant at the temperatures studied, and the narrow temperature range can be expected. The next step is an examination of an iron surface with TCP adsorbed onto it. A detailed examination of this type is currently in progress.

Conclusions

The following conclusions can be drawn from our study of the friction and wear of 440C, 52100, and M-50 steels lubricated with TCP, 3.45 percent TCP in squalene, and pure squalene as functions of temperature:

1. Tricresylphosphate (TCP) reacts with 52100 steel in dry air to form a protective film. This reaction begins at 220° C, the assignable characteristic temperature T_r ,

which is almost the same as the T_r value for TCP reacting with M-50.

2. TCP reacts with 440C steel in dry air to form a protective film but only after the adsorbed TCP film is ruptured. There is no assignable T_r value because the friction peaks at 227° C.

3. TCP reacts with M-50, 440C, and 52100 steels under limited lubrication conditions in dry air at 223° ± 5° C. The reaction temperature is independent of the steel composition.

4. The presence of 3.45 percent TCP in squalene has no discernible influence on friction as a function of temperature for M-50, 440C, or 52100 steels in dry air.

5. Squalene exhibits characteristic temperatures that are sensitive to the steel surface. The 440C steel shows no assignable T_r value, 52100 steel has a T_r of 205° C, and M-50 steel has a T_r of 180° C. The reaction with the surface is attributed to oxidation products of squalene generated at the tertiary hydrogen sites in the molecule.

6. TCP does not react with 440C steel in dry nitrogen; failure behavior was observed at 220° C. TCP-lubricated 52100 steel exhibits a sharp peak in friction in dry nitrogen. Squalene-lubricated 440C fails very early in dry nitrogen, at 70° C; M-50 steel fails at 170° C.

7. Wear volume measurements demonstrate that the lowest wear for limited TCP lubrication of 440C and 52100 steels is achieved in the 229° to 237° C temperature range.

8. Oxygen is essential for the reaction of TCP with both 52100 and 440C steels.

Lewis Research Center

National Aeronautics and Space Administration

Cleveland, Ohio, October 13, 1983

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1. Report No. NASA TP-2274		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle Mechanism of Lubrication by Tricresylphosphate (TCP)				5. Report Date February 1984	
				6. Performing Organization Code 506-53-12	
7. Author(s) Owen D. Faut and Donald H. Buckley				8. Performing Organization Report No. E-1846	
				10. Work Unit No.	
9. Performing Organization Name and Address National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135				11. Contract or Grant No.	
				13. Type of Report and Period Covered Technical Paper	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546				14. Sponsoring Agency Code	
15. Supplementary Notes Owen D. Faut, Wilkes College, Wilkes-Barre, Pa., and National Research Council - NASA Research Associate; Donald H. Buckley, Lewis Research Center.					
16. Abstract The coefficient of friction was measured as a function of temperature on a pin-on-disk tribometer. Pins and disks of 440C and 52100 steels were lubricated with tricresylphosphate (TCP), 3.45 percent TCP in squalene, and pure squalene. M-50 pins and disks were lubricated with 3.45 percent TCP in squalene and pure squalene. Experiments were conducted under limited lubrication conditions in dry (<100 ppm H ₂ O) air and dry (<20 ppm H ₂ O) nitrogen at 50 rpm (equivalent to a sliding velocity of 13 cm sec ⁻¹) and a constant load of 9.8 N (1 kg). Characteristic temperatures T_r were identified for TCP on 52100 steel and for squalene on M-50 and 52100 steels, where the friction decreased because of a chemical reaction between the lubricant and the metal surface. The behavior of squalene obscured the influence of 3.45 percent TCP solute on the friction of the system. Wear volume measurements demonstrated that wear was lowest at temperatures just above T_r . Comparing the behavior of TCP on M-50, 440C, and 52100 steels revealed that the TCP either reacted to give T_r behavior or produced initial failure in the temperature range 223 ± 5°C. The 440C steel yielded a peak in friction; the 52100 and M-50 steels had assignable T_r values in this temperature range. Oxygen was essential for the reaction of TCP with the metal surface.					
17. Key Words (Suggested by Author(s)) Friction Wear Lubrication TCP			18. Distribution Statement Unclassified - unlimited STAR Category 37		
19. Security Classif. (of this report) Unclassified		20. Security Classif. (of this page) Unclassified		21. No. of pages 12	
				22. Price* A02	

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